



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Re: Application of ISNARD et al.

Serial No: 10/520,903

Filed: 01/11/2005

For: PROCESS FOR THE (CO-)POLYMERISATION OF OLEFINS IN THE GAS PHASE

DECLARATION OF ANDREW DAVID BELL

I, ANDREW DAVID BELL, do hereby solemnly declare as follows:-

1. I am a British subject, residing at 3 Allée de la Garrigue, Parc Privé de Barqueroute, 13620 Carry-le-Rouet, France.
2. I graduated from Cambridge University UK in 1974 with an MA(Hons) degree in Chemical Engineering.
3. I am currently employed by INEOS Technologies at the Research and Technology Centre, Lavera, France. Since 1990 I have been working almost exclusively on the Innovene (formerly BP) fluidised bed process for manufacture of polyethylene. Initially this was in a commissioning role, leading a number of Licensed Innovene start-ups around the world. Since 2000 I have been involved in after-sales support to Licensees, and in the design and development of the Innovene process. I am also a current member of the Institution of Chemical Engineers.
4. I am familiar with the technical details concerning the US Patent Application Serial No. 10/520,903 filed on January 11th 2005.
5. I have read and am familiar with the cited art, US 4,316,825 A1 to Wirth, and titled "HIGH GREEN STRENGTH RUBBERS", and EP 0 180 420 to Durand et al. and titled "PROCESS FOR THE START UP OF POLYMERISATION OR COPOLYMERISATION IN THE GAS PHASE OF ALPHA-OLEFINS IN THE PRESENCE OF A ZIEGLER-NATTA CATALYST SYSTEM".
6. The present invention relates to a gas phase polymerization or co-polymerization process reactor which process comprises a pre start-up operation characterized in that, prior to the introduction of the catalytic system in the reactor, the reactor is subjected to a cleaning treatment comprising the steps of introducing into the reactor an alkane having from 4 to 8 carbon atoms, circulating said alkane across the reactor under pressure and elevated temperature, depressurizing and purging the reactor, and wherein the quantity of alkane used for the treatment is such that the alkane partial pressure is comprised between 25 and 95% of the saturated vapour

pressure of the said alkane under the temperature and pressure treatment conditions.

7. Wirth relates to "High green strength rubbers" which can be prepared by polymerization in a solution phase process.
8. The Examiner references Example 1 of Wirth as disclosing a rinsing step using hexane, and states that it would be "obvious" to apply this to a gas phase process (as taught by Durand et al.) to obtain the process of the present invention.
9. I believe that this is incorrect, and that, as included in Applicant's reply dated November 20th 2007, the skilled person would not combine the teachings in respect of the individual reactors and processes of Wirth and Durand because they relate to distinct processes.
10. In support of this, I note that the rinsing with liquid alkane as taught by Wirth is not applicable to a gas phase reactor system. In particular, the volume of an industrial reactor system would typically be 1000 – 1500m³. The practical aspects of filling the reactor and associated pipework, exchangers and compressor with very large volumes of hazardous alkanes at elevated temperature and pressure, and then circulating the alkane, rule out industrial application of a liquid rinse of a gas phase reactor system. Further, the reactor cannot normally be isolated from the associated large-bore pipework and even treatment by liquid rinsing smaller subsystems is therefore not practical.
11. In the Advisory Action dated December 4th 2007, the Examiner argues that the liquid used in Wirth would in any case have a vapour phase associated with it.
12. However, the present claims require the quantity of alkane used for the treatment to be such that the alkane partial pressure is comprised between 25 and 95% of the saturated vapour pressure of the said alkane under the temperature and pressure treatment conditions. Under these conditions, no liquid phase of the alkane can be present, and thus the rinsing with liquid alkane, even if there were a vapour phase associated therewith, is not in any way comparable or equivalent to the process as presently claimed. Moreover, the believed mechanism claimed for the benefits of the current invention, as described at page 3, lines 9-15 of the specification, is different from that one would associate with a liquid rinse.
13. In the Advisory Action dated December 4th 2007, the Examiner also argues that the Applicant should provide comparative data showing the criticality of the claimed partial pressures compared to the prior art.
14. In fact, it is not possible to provide comparative data with rinsing with alkane as taught by Wirth but applied to a gas phase reactor because, as noted above, it is not practical to use a liquid to rinse a gas phase reactor.

15. Not least for the above reasons, it is believed that the skilled person would not combine the rinsing step of Wirth with the gas phase process of Durand et al. to obtain the process of the present claims, which are therefore believed to be not obvious over the cited art.
16. In further support of the inventive step of the present application, Appendix 1 presents as Figures 1 and 2 experimental data showing the benefits of the cleaning treatment of the process of the present invention compared to a more conventional process start-up without a depressurization step.
17. The data presented in Figures 1 and 2 shows, respectively, the measurements from temperature probes located in the vicinity of the fluidization grid for a comparative example compared to one according to the present invention. Variations of such temperature measurements are known to correlate to the presence of agglomerates in the reactor.
18. Figure 1 shows the temperature measurements obtained in the hours after start-up of a polymerization reaction not according to the present invention. During this start-up, the catalyst bed in the reactor was fluidized and then the pressure increased to reaction pressure (approx. 23 bars) over the course of approximately 9 hours. During this time pentane was introduced into the reactor and its partial pressure increased to 2.5 bars. No depressurization was carried out after the introduction of pentane prior to the introduction of catalyst (and the pentane was maintained in the reactor at approximately this pressure throughout the run). The reaction was then started and although the temperature measurements shown in Figure 1 were initially fairly smooth, variations soon started to appear. Approximately 18 hours after start-up these variations were very agitated, varying frequently and significantly. Subsequently, agglomerates caused a reactor shut-down after less than 24 hours operation.
19. For comparison, Figure 2 shows the temperature measurements obtained in the hours after start-up of a polymerization reaction according to the present invention. The start-up was similar to the start-up described above, the catalyst bed in the reactor being fluidized and then the pressure increased. In this case, however, after the pentane partial pressure has been increased to approximately 2 bars (at a total reactor pressure of approx. 16 bars), the reactor was depressurized and the pentane purged therefrom, and the reaction pressure then built up to reaction pressure of 23 bars (with approx. 2.5 bars pentane). The temperature measurements shown in Figure 2 show a much lower variance (note that the scale is significantly different to Figure 1) showing much more constant temperatures. Figure 2 shows 48 hours operation, but in fact the process in this Example operated for more than 10 days without need to stop the reactor due to agglomerates formation.
20. These results are typical of those obtained from a number of polymerization runs. In fact, since the introduction of a cleaning step comprising introducing an alkane and subsequently depressurizing according to the process of the

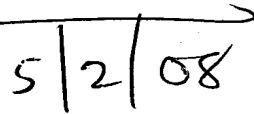
present invention the number of successful start-ups on the reactor used for the above examples has increased from about 50-70% to 100%.

All statements made of my own knowledge are true and all statements made on information and belief are believed to be true. I also understand that wilful false statements and the like are punishable by fine or imprisonment, or both (18 U.S.C. 1001) and may jeopardize the validity of the application or any patent issuing thereon.



ANDREW DAVID BELL

Date:



5/2/08